Quality Control of the Tribological Coating—PS212

Harold E. Sliney, Christopher DellaCorte, and Daniel L. Deadmore

Lewis Research Center

Cleveland, Ohio

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National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

SUMMARY

PS212 is a self-lubricating, composite coating that is applied by the plasma spray process. It is a functional lubricating coating from 25 °C (or lower) to 900 °C. The coating is prepared from a blend of three different powders with very dissimilar properties. Therefore, the final chemical composition and lubricating effectiveness of the coatings are very sensitive to the process variables used in their preparation. This document defines the relevant variables and describes process and analytical procedures that have resulted in satisfactory tribological coatings.

INTRODUCTION

The purpose of this paper is to present recommended procedures for the preparation of PS212 which is a multicomponent, plasma sprayed, solid lubricant coating. The quality of plasma sprayed coatings is very sensitive to details of spray powder preparation and to process variables such as target distance, gas flow rates, and electrical settings. This is especially so when spraying multicomponent powders. Quality control is further complicated for solid lubricant coatings because friction and wear characteristics are influenced by post-spraying finishing procedures. This paper describes procedures from powder preparation to final grinding that have been the most successful in the development of PS212 tribological coatings at NASA Lewis Research Center.

PS212 is a member of the PS200 series of composite coatings. These coating compositions were designed to provide low friction and wear over a large temperature spectrum. The prefix "PS" denotes that the coatings are applied by a "plasma spray" process. The three digit number identifies a specific coating formulation. Coatings in the "200" series contain by definition, a high percentage of nickel alloy-bonded chromium carbide for wear control, along with variable amounts of silver and a barium fluoride/calcium fluoride eutectic composition for lubrication. The last two digits are arbitrary and merely complete the identification of individual coating compositions.

In a study designed to optimize the proportions of the three primary constituents of the coatings, formulation number PS212 was the most effective lubricant in pin-on-disk friction and wear experiments (ref. 1). The nominal composition of this coating in weight percent is:

70 metal-bonded chromium carbide (Cr₃C₂)

15 silver (Aq)

15 barium fluoride/calcium fluoride (BaF₂/CaF₂) eutectic

Although this composition gave the lowest friction and wear in the optimization studies, different ratios of the three components may be better in certain types of application. For example, a higher carbide content may be preferred in high contact stress applications where higher compressive strength is important. The procedures described in this paper are specifically for PS212, but they also apply in general to other coatings in the PS200 series. However, details, especially of the plasma spray process, may differ for each member of the series.

A comprehensive listing of publications concerning the development and evaluation of the PS200 series of coatings at NASA Lewis is given in references 1 to 9. Reference 1, in particular compares various compositions in the PS200 series.

RECOMMENDED PROCEDURES

The scope of this section encompasses a description of procedures for: (1) the preparation of blended powders for plasma spraying; (2) the optimization of plasma spray parameters; (3) surface finishing by diamond grinding; (4) suggested quantitative chemical analytical procedures; (5) friction and wear characterization; and (6) optional heat treatment. Figure 1 is a flow diagram showing the sequence of these procedures. The feedback loops in this diagram illustrate the iterative steps that are used in developing a process that will result in coatings of correct chemical composition and acceptable morphology.

Powder Preparation

The chemical compositions and the particle size ranges of each of the three major components of PS212 are given in table I. The chromium carbide and silver powders are commercially available as plasma sprayable powders. The fluoride eutectic powder is prepared by mixing reagent grade (99.9 percent pure) powders of BaF2 and CaF2 in the eutectic ratio given in table I, then furnace melting them in a nickel crucible at 1100 °C in a nonoxidizing nitrogen atmosphere. The molten fluorides are held at temperature for about 1 hr, then moved to a cooling zone of the furnace to solidify. When cool, the solidified eutectic is removed from the crucible and crushed in a mechanical plate crusher into particles of about 1 to 2 mm in size. This coarse material is then ball milled with aluminum oxide pebbles into a fine powder. The powder is then sieved to obtain a particle size range of 44 to 74 μm . X-ray diffraction (XRD) analyses of these powders should be positive for the two fluorides only. Contamination levels high enough to be detected by XRD, are not acceptable.

The eutectic powder is then thoroughly blended with the carbide and silver powders. The powders are mixed by a vigorous shaking motion; rolling is not recommended because it can result in an undesirable agglomeration of the powder.

The moisture content of the powders influences their mixing and flow characteristics. The powders flow best when they are very dry. Therefore, only dry powders should be mixed and plasma sprayed. Blended powders should be stored in a desiccator or moisture-proof containers, and it is recommended that

they be further thoroughly dried by baking at about 125 °C just prior to plasma spraying. The superior flow characteristics of dried powders improves the deposit efficiency and the composition reproducibility of the coatings.

In summary, experience has shown that the plasma spray characteristics of the PS212 powder is strongly influenced by the powder preparation procedure. It is especially important to: (1) use prefused and repowdered eutectic rather than merely blended BaF_2/CaF_2 powders; (2) employ the particle size ranges given in table I; (The use of powders with different particle sizes may have a significant effect on coating characteristics); and (3) use thoroughly dry powders.

Optimization of the Plasma Spray Parameters

The next step after powder preparation is plasma spraying. The plasma spray process is shown schematically in figure 2. In principle, the process is simple: an inert gas is electrically ionized and accelerated to produce a high-speed extremely high-temperature plasma; the powders to be sprayed are injected into the plasma where they partially or completely melt, then impinge on the substrate where they solidify and form an adherent coating. In practice however, the process is complicated because it involves many significant variable parameters. These include, for example: ionization voltages and currents, ionization and carrier gas flow rates and pressures, plasma spray gun design, gun to substrate distance, substrate temperature, and powder flow characteristics.

Stainless steel and nickel alloys are the preferred substrates for PS212. The surface to be coated is first sand blasted and then plasma sprayed with a nichrome bond coat about 0.08 mm thick. The PS212 lubricant coating is then applied.

Typical plasma spray parameters which have produced satisfactory coatings of PS212 are given in table II. It should be stressed, however, that parameters must be established for each plasma spray facility and coating, especially when multicomponent powders are being sprayed. Therefore, a systematic method was developed to optimize the process. The steps in this method are included in figure 1, and essentially consist of an iteration of spraying and analyses. A set of test coupons is sprayed using a range of spraying parameters around those listed in table II. This is followed by microscopic examination and chemical analyses of the coatings. The results are used as a guide to any further parameter adjustments that may be necessary. The process is repeated until the chemical composition of the coatings is within the limits given in table III. The microporosity, which is characteristic of plasma spray coatings, is acceptable; but gross voids detectable by low power, e.g. x20, microscopy, are not acceptable. A SEM micrograph of a coating cross section illustrating the distribution of coating components is given in figure 3(a). A comparison of unacceptable and acceptable levels of porosity is given in figure 3(b).

After parameter settings are found which produce satisfactory test coupons, they are used to spray friction and wear test specimens (tribotest specimens).

Surface Finishing (Grinding Procedure)

The coatings are sprayed to a thickness of about 0.35 mm and then diamond ground to the desired thickness (usually 0.25 mm including the thickness of the bond coat) and a surface finish that is suitable for a sliding contact bearing or seal application. The following grinding procedure has consistently produced satisfactory results.

- (1) Use diamond grinding or cubic boron nitride grinding only.
- (2) Use water as the only grinding fluid (no oil).
- (3) Initial grinding depth should be 0.025 mm (0.001 in.).
- (4) Final cuts should be 0.010 to 0.015 mm (0.0004 to 0.0006 in.).
- (5) Dress the wheel often with a diamond dressing tool or diamond matrix stick.
 - (6) Use low feed rates to minimize grinding marks.
- (7) Direct the water spray into the grinding contact area to continuously wash particles away.
- (8) The final surface should be smooth, but have a speckled appearance (due to the composite nature of the coating).

The ground surfaces should be examined macroscopically to insure that the grinding did not smear or crack the coating surface. These defects are indications that either the wheel was not dressed in a manner that makes a clean cut or the depths of cut were incorrect. Figure 3(c) is a low magnification photomicrograph of a diamond ground surface of PS212 that is considered acceptable. No cracks or large voids are present, and the soft silver and fluorides have not been selectively removed by the finishing operation. It is difficult to specify an acceptable surface finish for the coatings because of the micropores at the surface. The coatings are ground in a manner that produces surfaces that are smooth in terms of asperity size, but nevertheless have a high root mean square surface finish because stylus profilometry averages micropores as well as asperities. The ground surfaces typically have a 0.7 to 1.0 μm finish, but this is reduced to 0.1 to 0.2 after a brief duration of sliding which closes the surface pores. Incorrect finishing procedures can alter the chemical composition of the ground surface. For example, if the bulk composition of the coating is correct, but the surface is deficient in silver or fluorides, it indicates that the finishing procedure has selectively removed the softer components of the coating. This should not occur if the coatings are ground properly.

Quantitative Chemical Analyses

Table III gives the ranges of elemental composition that have given acceptable performance in PS212. Our experience has shown that the silver content and the fluoride eutectic content (computed from the barium and calcium

content) may vary as much as plus or minus 20 percent of the nominal, (in other words 15±3 percent) without adversely affecting coating performance. Wet chemical analyses are useful for determining the average bulk composition of the coatings, and are used to establish standards or to check the accuracy of instrumental analytical techniques. However, the near surface region, sometimes called the surficial region, contains the material volume that is of most tribological importance, and this region does not necessarily have the same composition as the bulk. An example of this is the depletion of some coating components by an incorrect finishing operation.

An energy dispersive (EDS) x-ray technique, which is used in conjunction with many SEM installations, can be a useful analytical tool for plasma sprayed coatings. The primary excitation source is a 15 keV electron beam which excites characteristic x-ray emission of the elements. The depth of analyses is on the order of l μm . This relatively shallow sampling depth is an advantage in differentiating surficial composition from bulk composition. The disadvantage is that thin contaminant films such as an oxide tarnish on the surface can give misleading results. Therefore, surfaces must be free of visible surface films during the analyses. Elemental quantitative analysis programs are available for use with EDS. We use a Princeton Gamma Tech EDS unit with a program called NOSTD which does not require calibration standards, but computes ZAF corrections for each element to correct for atomic number (Z), x-ray absorption (A), and fluorescence (F).

Another technique for analyzing these coatings is x-ray fluorescence (XRF). XRF provides about one order of magnitude greater depth of analysis than EDS. This reduces the possibility of misleading results caused by thin contaminant films while still detecting deficiencies of any of the major components in the surficial region. Clearly, an important problem of chemical analyses is to choose an optimum depth of analyses. The subject of depth of analysis by various analytical techniques is thoroughly discussed in reference 10. XRF is an attractive technique for analyzing plasma spray coatings for a number of reasons in addition to depth of analysis considerations. For example, XRF can be done in room air thus eliminating the complexity of a vacuum system. Further, portable XRF systems that can accurately analyze plasma sprayed coatings immediately following their application, are available at a relatively modest cost. These portable instruments use isotope radiation to stimulate characteristic x-rays of the elements in the sample. Peak heights of the spectra for the sample are then compared to those of standards of known composition. Portable XRF instruments are generally simple to use, reliable, and can provide coating analyses in a few minutes once suitable standards have been programmed into the instrument. Because of the rapid analysis capability and portability, these instruments are very useful in optimizing plasma spray parameters as well as for verifying coating composition from batch to batch.

Image analyses microscopy has been used to estimate area percentages of distinguishable components in other composite coatings (ref. 11). It may be possible to analyze PS212 by this method. Metallographically polished specimens are required. If the assumption is made that area percentages represent actual component ratios, they can be considered to be equal to the volume percentages. Table IV gives the volume percentages of the components in PS212 calculated from their weight percentages and densities.

Friction and Wear Characterization

The self-lubricating quality of the coatings is evaluated by doing friction and wear tests using a bench test machine such as a rub block on a disk or a pin-on-disk device. Test specimen configurations for these devices are shown in figure 4, and a sketch of a pin-on-disk tribometer is shown in figure 5. A cobalt base alloy, Stellite 6B with a hardness of Rockwell C-40, is a preferred counterface material for the PS200 series of coatings.

Table V from reference 5 gives the friction coefficients and wear factors that have been measured in pin-on-disk tests of Stellite 6B sliding on PS212 in atmospheres of helium, or air at temperatures of 25, 350, and 760 °C. Corresponding data for PS212 in hydrogen were not measured but the friction and wear of PS200 which has a similar composition (80 metal-bonded $Cr_3C_2-10A_5-10$ fluoride eutectic) are included in table V for reference. The wear factors are given in units of cubic millimeters wear volume per Newton load per meter sliding distance (mm³/Nm). The cylindrical pins are 9.5 mm in diameter with a 4.75 mm spherical tip that slides against the flat, coated surface of a rotating disk to form a circular wear track of 16 cm circumferance on the coating. The normal load is 0.5 kg, and the sliding velocity is 2.7 m/s. Coatings evaluated under similar conditions should have friction and wear values that do not substantially exceed those given in table V. Higher friction coefficients indicate that the chemical composition in the surficial region is probably wrong due to either incorrect plasma spray procedures, or incorrect grinding.

Optional Heat Treatment

PS212 can be heat treated to cause fluorides to migrate from the interior of the coating to the surface. A heat treatment consisting of 2 hr at 900 °C in a hydrogen atmosphere, increases the fluoride content of the surface layer and improves the friction and wear properties. (See figs. A-1 and A-2 in appendix I.) Table VI gives the friction and wear data in air for heat treated PS212. A comparison with the data in table V for PS212 that was not heat treated shows that both friction and wear in air are reduced by this treatment.

The heat treatment procedure was not optimized. Shorter firing times for example may be necessary to avoid excessive fluoride buildup on surfaces where small clearances are required.

CONCLUDING REMARKS

- 1. The procedures outlined in this paper, when accurately followed, will reproducibly produced PS212 coatings with good tribological properties over a large temperature range.
- 2. The quality of PS212 plasma spray coatings is sensitive to the details of preparation, especially in regard to the plasma spray parameters and the finishing procedure.
- 3. A cobalt-base alloy, Stellite 6B, is the best counterface alloy found to date for sliding against PS212.

4. It is the intent of this document to assist in the transfer of technology developed at NASA to U.S. industries.

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TABLE I. - NOMINAL COMPOSITIONS AND PARTICLE SIZES OF THREE MAJOR COMPONENTS USED IN PREPARING PS212

Component	Composition,	Particle size			
	wt %	U.S. sieve number	Micrometer range		
Metal-bonded chromium carbide: Cr3 ^C 2 Ni	 58 28	-200 + 400	74 to 37		
Co Al Silver (Ag) Eutectic: BaF ₂ CaF ₂	12 2 100 68 32	-100 + 325 -200 + 325	149 to 44 74 to 44		

TABLE II. - TYPICAL PLASMA SPRAY PARAMETERS

[Must be optimized for each facility. Equipment: Plasmadyne SG100 gun; anode/cathode pair no. 155; single powder injection port; gas injector no. 110; auxiliary gas, helium.]

Arc gas			Argon
Arc gas flow rate, m ³ /hr			. 1.4
Powder carrier gas			Argon
Powder carrier gas flow rate, m ³ /hr			. 0.4
Coating powder flow rate, kg/hr			1
Amperage, A		400	to 475
Voltage, V			. 32
Gun to specimen distance, mm			~150

TABLE III. - COATING COMPOSITION GUIDE

(a) Allowable composition scatter in PS212 coating components

C ompo nent	Scatter, wt %
Metal-bonded	70±6
Cr ₃ C ₂ Ag BaF ₂ /CaF ₂ eutectic	15±3 15±3
(62/38 weight ratio)	

(b) Elemental compositions of coatings

Element ^a	Nominal composition of metal-bonded Cr ₃ C ₂ , wt %	Composition range for PS212, wt %	Elements analyzed by EOS or XRF
Cr C Ni Co Al Ag Ba Ca F	48 6 28 12 2 0	33.6±3.0 4.2 (nominal) 19.6±2.0 8.4±0.8 1.4±0.2 15.0±3.0 7.3±1.5 2.9±0.6 4.8±1.0	X (b) X X X X X (c)

^aBalance: Mo, B, and Si. ^bNot analyzed. Computed as 70 percent of carbon in metal-bonded chromium carbide. ^cNot analyzed. Computed from analyses for Ba and Ca.

TABLE IV. - NOMINAL VOLUMETRIC COMPOSITION OF PS212

[Theoretical average density: $\bar{\sigma} = 6.60 \text{ gms/cm}^3$.

Component	Density, gm/cm ³	Content in PS212, vol %
Cr ₃ C ₂	6.68	37.2
Ni alloy binder Ag BaF ₂ CaF ₂	10.50 4.80 3.18	Balance 9.4 11.8 12.7

TABLE V. - FRICTION AND WEAR OF PS212 IN AIR AND IN HELIUM AND OF PS200 IN HYDROGEN

[Test conditions; Stellite 6B pins of 4.76 hemispherical radius sliding on coated disks; load, 4.9 N; sliding velocity, 2.7 m/s.]

Coating	Test atmosphere	Temper- ature, °C	Friction coefficient, µm	Pin wear factor, ^a K, mm ³ /N m	Coating wear factor, K, mm ³ /N n
P\$212	Air	760 350	0.40±0.02 0.36±0.01	3.2×10 ⁻⁶	2.5×10 ⁻⁵ 1.6
P\$212	Helium	25 760 350	0.28±0.01 0.26±0.02 0.25±0.03	.7 6.2×10 ⁻⁷ 2.8	5.6 1.8 .4
PS200	Hydrogen	25 760 350	0.24±0.02 0.18±0.05 0.26±0.06	5.2 .75 1.25	.2 7.6×10 ⁻⁶ 4.5
		25	0.26±0.04	2.0	7.0

 $a_{K} = \frac{\text{Wear volume}}{\text{Load} \times \text{Sliding distance}} = \frac{\frac{3}{\text{mm}}}{\text{N m}}$

 $K < 10^{-6}$ indicates low wear;

 10^{-6} < K < 10^{-5} indicates moderate wear;

 $K > 10^{-5}$ indicates severe wear.

TABLE VI. - FRICTION AND WEAR OF HEAT-TREATED PS212 SLIDING ON STELLITE 6B IN AIR

[Heat treatment: 900 °C for 2 hr in hydrogen. Test conditions: 4.76-mm spherical-radius Stellite 6B pin on PS212 flat; load, 4.9 N; sliding velocity, 2.7 m/s.]

Temper-	Friction	Pin wear	Coating wear
ature,	coefficient,	factor, ^a	factor, a
°C	µm	mm ³ /N mx10 ⁶	mm ³ /N mx10 ⁶
76 0	0.29±0.02	(2.2±0.3)×10 ⁻⁶	1.3×10 ⁻⁶
35 0	0.28±0.01	(0.5±0.1)	.5
25	0.16±0.01	(2.0±0.6)	3.2

^aPin wear was measured every 20 min during a 60-min wear test. Disk wear was measured once after each 60-min wear test.

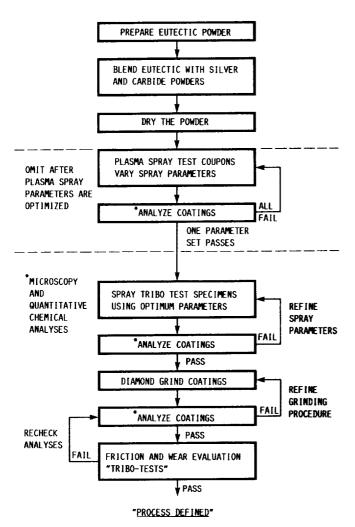


FIGURE 1. - PROCESS FLOW CHART FOR OPTIMIZING THE PREPARATION OF PS212 COATINGS.

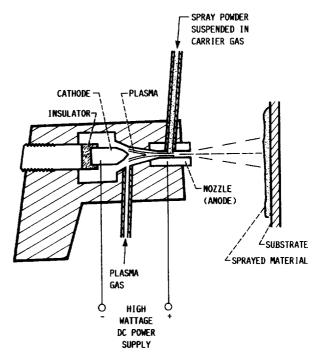


FIGURE 2. - SCHEMATIC CROSS SECTION OF BASIC COMPONENTS OF PLASMA SPRAY GUN (WATER COOLING CHAMBERS OMITTED TO SIMPLIFY SCHEMATIC).

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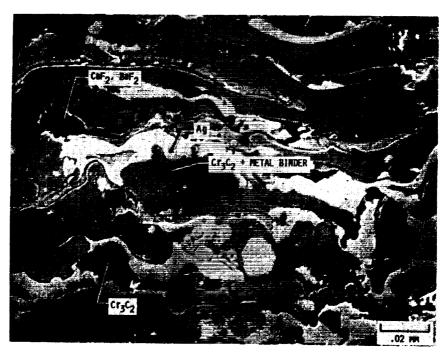
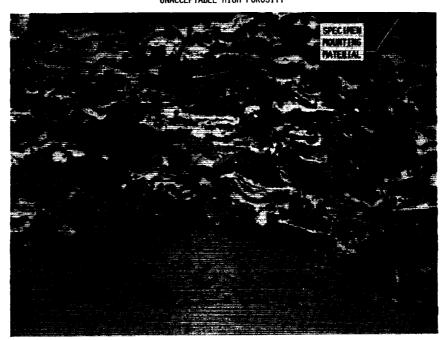


FIGURE 3A. - SEM CROSS SECTION OF PS212 SHOWING DISTRIBUTION OF COMPONENTS INFERRED FROM ELEMENTAL ANALYSES BY EDS. ORIGINAL MAGNIFICATION OF 600X.

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UNACCEPTABLE HIGH POROSITY



ACCEPTABLE POROSITY

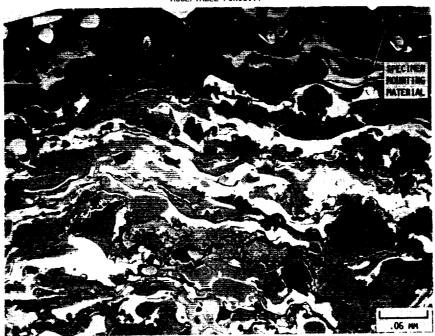


FIGURE 3B. - CROSS SECTION OF UNGROUND PLASMA SPRAYED PS212 COATING ILLUSTRATING UNACCEPTABLE AND ACCEPTABLE DEGREE OF POROSITY. ORIGINAL MAGNIFICATION OF 200X.

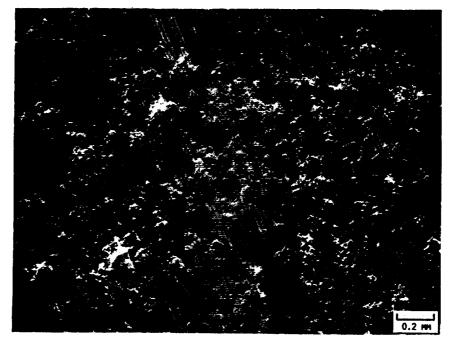


FIGURE 3C. - SEM OF DIAMOND GROUND PS212 ORIGINAL MAGNIFICATION OF 50X.

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FIGURE 4. - FRICTION AND WEAR TEST-SPECIMEN CONFIGURATIONS.

(B) PIN ON DISK.

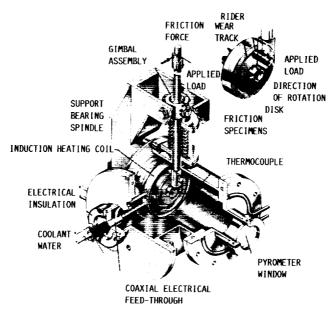


FIGURE 5. - HIGH-TEMPERATURE FRICTION APPARATUS.

APPENDIX I

ANALYSIS OF PS212 SURFACES BY EDS TO SHOW EFFECT OF HEAT TREATMENT

ON SURFACE COMPOSITION

Figure A-1(a) is an EDS incremental area scan along one radius of a 6.4-cm diameter disk coated with PS212 and finished by diamond grinding. Each of 11 equally spaced area increments along a disk radius was analyzed to obtain the radial composition distribution of the coating. The composition is seen to be quite uniform over the entire radius. However, the calcium and barium concentration are very low. It is believed that the soft calcium and barium fluorides were removed by incorrect grinding.

An experiment was then performed in which the disk was induction heated for 2 hr in hydrogen to produce a temperature gradient from 905 °C at the rim to 800 °C at the edge of the center hole of the disk. Figure A-1(b) shows minimal concentration changes relative to the untreated disk on the surface near the center of the disk which was heated to 800 °C, but very large changes relative to the untreated disk as the 900 °C region is approached. In the region heated to about 900 °C, a very large increase in barium and calcium is observed. Silver concentration is correspondingly diluted by this large increase of barium and calcium. This technique not only identified composition change, but in one heat treatment, identified the temperature level necessary to achieve it. The heat treated disks gave superior tribological performance that we attribute to calcium and barium fluoride enrichment of the surface. The reduced friction and wear of heat treated PS212 was discussed in the main body of the text. Compare tables V and VI.

If the coating is deficient in calcium and barium fluoride throughout its thickness (and not only at the surface due to incorrect grinding), the heat treatment of course produces no beneficial effect. This is illustrated in figures A-2(a) and (b). EDS analyses along the radius of a coating that was deficient in calcium fluoride and barium fluoride because of incorrect plasma spray parameters shows that only very small increases in barium and calcium are achieved by the heat treatment. These coatings were poor tribological in both the as-ground and the heat treated condition.

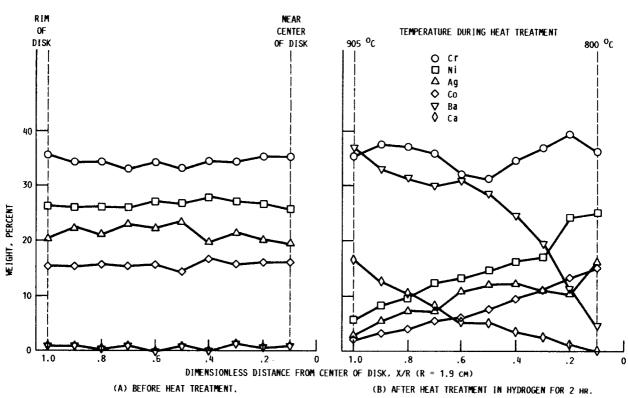


FIGURE A-1. - RADIAL COMPOSITION DISTRIBUTION OF A PS212 COATING DEFICIENT IN SURFACE CaF₂ AND BaF₂ AFTER GRINDING.

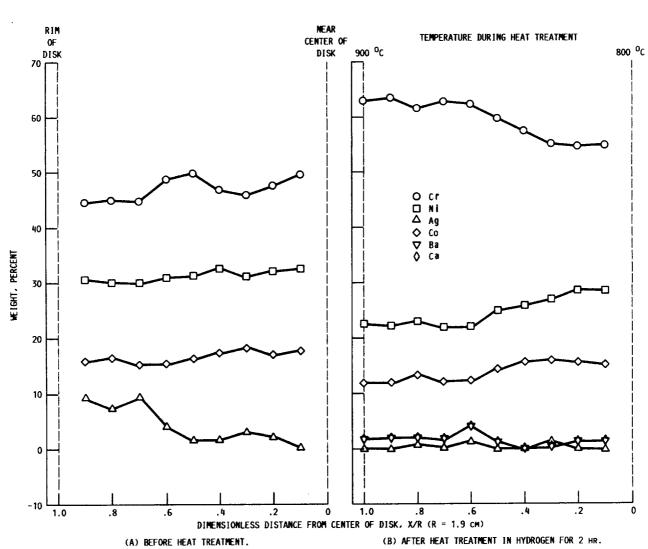


FIGURE A-2. - RADIAL COMPOSITION DISTRIBUTION OF A PS212 COATING DEFICIENT IN CaF₂ AND BaF₂ THROUGHOUT ITS THICKNESS.

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